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HEAT RESISTANT EXPLOSIVES XXII. THE PREPARATION OF 2,2',2'',2''',4,4',4'',4''', 6,6',6'',6'''-DODECANITROQUATERPHENYL, DODECA. (C)

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UNITED STATES NAVAL ORDNANCE LABORATORY, WHITE OAK, MARYLAND

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HEAT RESISTANT EXPLOSIVES XXII. THE PREPARATION OF 2,2',2'',2''',4,4',4'',4''',6,6',6'',6'''-DODECANITROQUATERPHENYL, DODECA. (C)

by

J. C. Dacons

ABSTRACT: The compound 2,2',2'',2''',4,4',4'',4''',6,6',6'',6'''-dodecanitroquaterphenyl, DODECA, has been prepared in 70% yield by the treatment of 3-chloro-2,2',4,4',6,6'-hexanitrobiphenyl, PIPICL, with copper bronze in nitrobenzene at 190°C. When purified by recrystallization from mixed solvents, the product was chromatographically pure and showed no tendency to melt at 450°C. The impact sensitivity and vacuum stability indicate that it is a potentially valuable heat resistant explosive. The cost of manufacture will probably be less than that of NONA. (C)

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This report describes the synthesis of 2,2',2'',2''',4,4',4'',4''',6,6',6'',6'''-dodecanitroquaterphenyl, DODECA. Vacuum stability and impact sensitivity tests indicate that this compound is a potentially useful heat resistant explosive suitable for use in mild detonating fuses, MDF, flexible linear shaped charges, FLSC, and other heat resistant cutting devices. The compound is a member of the polynitropolyphenyls derived from 1,3,5-trinitrobenzene. This work was carried out in part under the Study of Explosives Properties, Task RMMO 62 058 F008 08 11 Problem 012, and in part under Desensitization of Explosives, Task RMMO 62 058 F008 08 11, Problem 007.

J. A. DARE
Captain, USN
Commander

Albert Lightbody
ALBERT LIGHTBODY
By direction

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INTRODUCTION

In 1960 Dacons, Kamlet and Sickman (1) reported that the decomposition of TNT at elevated temperatures was largely by an oxidative attack on the methyl group by neighboring and non-neighboring nitro groups. It was therefore suggested that compounds containing nitro groups along with easily oxidized substituents would be basically unstable at elevated temperatures. The above study was made at 200°C, far above the melting point of TNT and any generalization of the findings would be strictly applicable only to compounds in the molten state or in solution in inert solvents. Stability of this type, frequently referred to as intrinsic stability, is due to the structure and content of the molecule and is unaffected by the energy of the crystal lattice. Several high energy compounds containing oxidizing nitro groups as well as oxidizable substituents are useful heat resistant explosives mainly by virtue of their high melting points. Notable among such explosives are HNS, DATB and DIPAM, and in the latter two the oxidizable amino groups contribute greatly to the elevation of the melting points of the basic trinitrobenzene and hexanitrobiphenyl structures. The effects of several oxidizable substituents in lowering the thermal stability of polynitro aromatic molecules has been confirmed by Rosen et. al. (2) who studied the vacuum stabilities of 1,3,5-trinitrobenzene, TNB, solutions of several compounds containing such substituents.

It has long been known that TNB is stable to temperatures as high as 300°C (3,4). However, its low melting point, 121°C, severly limits its usefulness as a heat resistant explosive. The TNT study led to the prediction that compounds such as 2,2',4,4',6,6'-hexanitrobiphenyl,HNB, 2,2',2'',4,4',4'',6,6',6''-nonanitroterphenyl, NONA, and 2,2',2'',2''',4,4',4'',4''', 6,6',6'',6'''-dodecanitroquarterphenyl, DODECA, would have good thermal properties and would melt high enough to be of practical value. However, it was observed that as the polymer-homologous series was ascended, the number of crowded centers between the rings increased and it was felt that the resulting steric effects would detract from the intrinsic stability of the molecules as they grew larger.

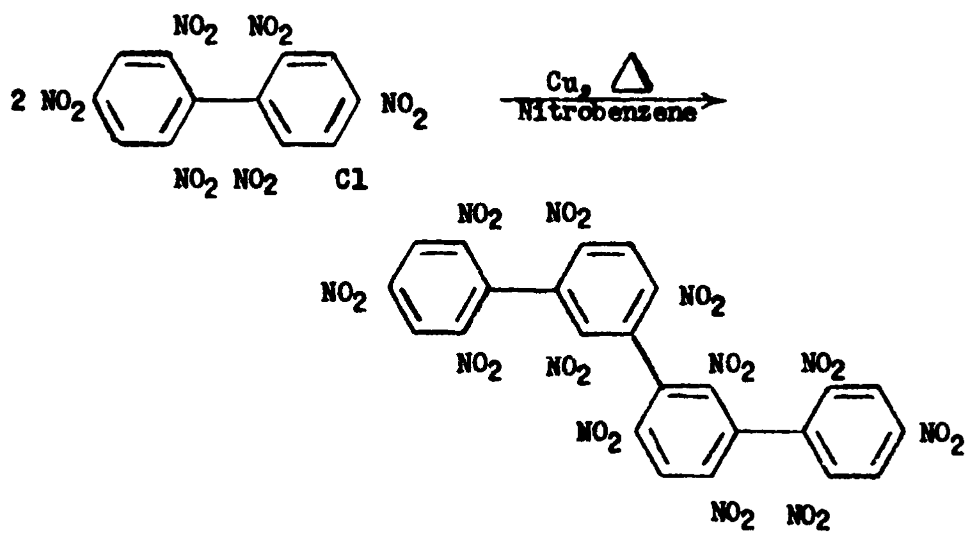
Hexanitrobiphenyl is a known compound (5,6) but no record of its having been evaluated for use at elevated temperatures could be found. A sample of this compound was prepared by the method of Ullmann, and a sample of NONA was prepared from picryl chloride and 1,3-dibromo-2,4,6-trinitrobenzene via a mixed Ullmann reaction in nitrobenzene (7). It was found that the thermal stability of HNB, mp 241-2°C, either in the molten state

or in TNB solution at 260°C was surpassed only by that of TNB. At the same time, it was found that the solution thermal stability of NONA, which melts in the range of 440-450°C, was slightly lower than that of HNB at 260°C, but that its stability in the solid state at this temperature was unsurpassed. Unfortunately, the usefulness of HNB is limited by phase changes at 128-130°C (8). On the other hand, NONA shows promise of becoming an extremely valuable high temperature explosive and an improved procedure for its preparation has been developed (9).

The nonanitroterphenyl having been prepared, evaluated, and a reasonable procedure for its large-scale production developed, our interest then shifted to DODECA, the next member of the series. The objectives of this research were to prepare enough DODECA for a preliminary evaluation of its thermal properties and sensitivity, and, should these prove interesting to develop a reasonable procedure for its large-scale production. This report describes the work done in efforts to achieve these objectives.

RESULTS AND DISCUSSION

The series of polynitropolyphenyls derived from 1,3,5-trinitrobenzene, TNB, has been extended to include 2,2',2'',2''', 4,4',4'',4''',6,6',6''6'''-dodecanitroquaterphenyl, DODECA. This compound was prepared from 3-chloro-2,2',4,4',6,6'-hexanitrobiphenyl, PIPICL, via an Ullmann reaction in nitrobenzene at 190°C*. The yields were about 70% of recrystallized,



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*The starting material, PIPICL, may be prepared from m-bromoanisole according to the three-step procedure of Dacons and Kamlet (10). The details of this synthesis are not given in this report.

chromatographically pure product. After filtering to remove the inorganic residue, the solvent was removed from the crude DODECA by steam distillation. The dark brown solid was digested in refluxing methanol to remove methanol-soluble impurities which included small amounts of starting material and HNB. The latter was probably formed by the dehalogenation of PIPICL caused by the presence of traces of water or other active hydrogen compounds during the reaction. The fact that small amounts of dehalogenation often occur during Ullmann reactions, even though special efforts are made to purify and dry starting materials, may indicate that compounds of the above type are formed as a result of side reactions.

The crude product was recrystallized by dissolving in acetone, adding a higher boiling solvent in which DODECA is only slightly soluble, and distilling until most of the acetone had been removed. Acetonitrile, benzene, toluene and glacial acetic acid have been used as the second solvent. Using vacuum stability and bulk density of the product is criteria, the latter two solvents gave the better results. As in the case with NONA, the variation in the solubility of DODECA with temperature in the solvents used is very slight. This eliminates the possibility of dissolving it in a hot solvent and effecting crystallization by cooling. A point of interest is the relative solubilities of NONA and DODECA. Although the molecular weight of the latter is approximately 33% greater, it is considerably more soluble in common solvents. For this reason, the procedure used for the recrystallization of NONA, i.e., the concentration of its acetone solutions until crystallization occurs, cannot be used for DODECA.

For vacuum stability, impact sensitivity and bulk density tests, samples of DODECA were purified by recrystallization from the above solvents pairs and dried at 150°C for sixteen hours. The results of these tests are given in Table 1. The vacuum stability tests were run at 260°C and the 20-minute surge represents the volume of gas per gram of explosive evolved during the first twenty minutes of heating. High values for this period often reflect incomplete solvent removal. The gas evolved thereafter is given in volume per gram per hour. Results are given for two hour periods and for twenty four hour periods. For comparison, the results for a purified sample of NONA under the same conditions were 0.43 cc/g for the 20-minute surge, 0.16 cc/g/hr and 0.18 cc/g/hr for a two and a nineteen hour period, respectively. Purified samples of DODECA and NONA were heated at 260°C under vacuum stability conditions for 78 hours and the residues were analyzed by thin-layer chromatography according to the procedure of Hoffsommer (11). The results indicated that the decomposition of DODECA amounted to 57% as compared to 35% for NONA. The difference is considerably less than would be suspected from the gas evolution rates given in Table 1.

The impact sensitivity of DODECA places it in the class with tetryl, HNS-I and NONA. Its bulk density, except for the sample recrystallized from acetone-acetonitrile, was slightly higher than HNS-I. It is interesting to note that the compound crystallizes as very thin, fragile, hexagonal plates which tend to break up on handling. The sample having the most perfectly formed crystals as well as the largest particle size was that from acetoneacetonitrile. Ironically, this sample showed the lowest bulk density of those tested. However, all of the above samples poured well through a glass powder funnel having a bore of approximately 5/16 inch. If they were not deliberately crushed, they appeared to have no static charge and there was very little tendency toward dusting.

These preliminary results indicate that DODECA is potentially a valuable heat resistant explosive which may be useful in the space program with its numerous and varied requirements for explosives of this type. Although somewhat less thermally stable than NONA, it ranks far better in this respect than most explosives in the heat resistant class. Pending engineering tests for specific applications, it appears that DODECA may be useful for such applications as high temperature FLSC, MDF and other cutting devices. A choice between it and NONA for some applications may ultimately be made on the basis of manufacturing costs. The synthesis of DODECA from commercially available materials, involves a four-step procedure, whereas that of NONA requires only two steps. The actual reaction time for the NONA synthesis is more than twice that for the DODECA synthesis. However, the most time-consuming step in the preparation of NONA, when done on a laboratory scale, may be carried out over night without attention. The overall yields of DODECA from the four-step synthesis are nearly twice those of NONA and the isolation is much simpler and less likely to introduce problems. The starting materials for NONA are picryl chloride and _m_-dibromobenzene whereas those for DODECA are picryl chloride and _m_-bromoanisole. Although the prices of these compounds appear not to be firmly established, they are relatively high and probably depend to some extent on demand. The yields in the NONA synthesis are based on _m_-dibromobenzene and a large excess of picryl chloride is required. The excess of the latter reactant is converted to HNB, an explosive which has not as yet proved to be useful. Although the DODECA synthesis requires the use of a large excess of _m_-bromoanisole as a solvent, most of it remains unchanged and can be recovered. The preparation of one pound of NONA requires approximately five pounds of picryl chloride and two pounds of _m_-dibromobenzene. The DODECA synthesis is considerably more economical with respect to costly organic starting materials. Assuming that 70% of the excess of _m_-bromoanisole is recovered, the preparation of one pound of DODECA would require about 1.5 pounds each of the anisole and

TABLE 1

Physical Properties of DODECA

Recrystallization Solvent	Vacuum Stability at 260° /a 20-min Surge cc/g	2-hr Period cc/g/hr	24-hr Period cc/g/hr	Bulk /b Density g/cc	Impact Sensitivity /c 50% Height, cm	Sigma
Acetone-Toluene	0.43	0.58	0.54	0.28	31	0.17
Acetone-Benzene	0.64	0.67	0.74	0.31	--	--
Acetone-Acetonitrile	0.90	0.87	0.85	0.16	--	--
Acetone-Acetic Acid	0.47	0.44	0.37	0.26	35	0.31

/a H.T.Simmons, Sr., NOLTR 65-206, in process.
/b R.E.Oesterling and J.C.Dacons, NOLTR 64-102, 9 July 1964.
/c NOL modified ERL Machine using 2.5 kg wt. on sandpaper.

picryl chloride. Since the cost of solvents and inorganic reagents would be small compared to the above materials, it is obvious that the materials cost for the synthesis of a quantity of DODECA would be considerably less than those for the synthesis of an equal quantity of NONA.

ACKNOWLEDGEMENTS

The author wishes to thank Dr. J. C. Hoffsommer for the quantitative thin-layer chromatographic analyses and Mr. H. T. Simmons, Sr. for vacuum stability determinations. Impact sensitivities were determined by Mrs. Sarah F. Duck of the Chemical Engineering Division.

EXPERIMENTAL

A solution of 118 g (0.257 moles) of PIPICL in 400 ml of dry nitrobenzene was made in a 1000 ml 3-neck round bottom flask fitted with a mechanical stirrer, powder funnel and thermometer. The temperature was raised to 180°C on an oil bath and 12 g of copper bronze* was added in one portion with

*Venus Natural Copper Fine 44-F, United States Bronze Powder Works, Flemington, N. J. The order of addition may be varied in that a slurry of copper bronze may be made in the reaction flask and a solution of PIPICL in nitrobenzene added through an addition funnel.

vigorous stirring. The temperature was raised to 190°C over a period of seven minutes. At this temperature, the copper began to lose its metallic luster. Starting approximately five minutes after the first copper addition, an additional 28 g of copper was added in four portions over a 15-minute period. The reaction was slightly exothermic and the temperature rose to 195°C during the addition. After the addition was complete, the temperature was held at about 190°C for 15 minutes after which the heating bath was removed. The mixture was allowed to cool to about 100°C and filtered with suction through a fiber glass filter. The solid inorganic residue was washed with acetone until the filtrate was nearly colorless. The solvent was then removed from the filtrate by steam distillation, after which the aqueous layer was removed by decantation. The dark solid product was then digested for 30 minutes in 1000 ml of refluxing methanol. The mixture was then filtered and the product was thoroughly washed with additional methanol. After drying in a warm oven, the light brown solid weighed 94 g. Thin-layer chromatographic analysis on Silica Gel G, using benzene as the developer, indicated that the product was mainly

DODECA but contained a trace of HNB and a larger quantity of an apparently more polar material which remained at the origin. A chromatogram of the methanol extract indicated the presence of an appreciable amount of HNB, a minute trace of DODECA and some material which remained at the origin.

The crude DODECA was dissolved in 2000 ml of hot acetone, treated with 50 g of Darco G-60, filtered and the carbon pad was washed with acetone. The filtrate, which was still dark, was concentrated by distillation on a steam bath with moderate stirring until precipitation just started. A volume of 500 ml of toluene was then added through an addition funnel, as the distillation continued, at about the same rate as that at which the distillate was removed. When distillation on the steam bath ceased, the bath was removed and the mixture was allowed to stand for two hours. It was then filtered, the product was thoroughly washed with toluene and dried in a warm oven. The yield was 76 g (70%) of a cream colored finely crystalline product. It showed no tendency to melt at 450°C, but began to darken in the range 300-350°C. Thin-layer chromatographic analysis indicated that it was pure DODECA.

Anal. Calcd. for $C_{24}H_6N_{12}O_{24}$: C, 34.1; H, 0.71; N, 19.8. Found: C, 33.85, 33.96; H, 0.66, 0.99; N, 19.17, 19.39.

An attempt to obtain a second crop from the mother liquor by distilling on a heating mantle until the temperature of the distillate was 110°C resulted in the recovery of about 1 g of dark brown solid which was shown by TLC to contain only a trace of DODECA.

Recrystallization of DODECA. In efforts to obtain maximum purity and higher bulk density, samples of the above product, or similar products, were recrystallized a second time from four solvent combinations as described below.

Recrystallization from Acetone-Toluene. A 21.5 g sample of the above once recrystallized product was dissolved in 300 ml of acetone, treated with 15 g of Darco G-60, filtered with suction through a fiber glass filter and the carbon pad was washed with 100 ml of acetone. The amber colored solution was distilled on a steam bath with moderate stirring until 100 ml of solvent had been removed. Then, as slow distillation continued, 150 ml of toluene was added through an addition funnel at approximately the same rate as that at which the distillate was removed. When 75 ml of toluene had been added, the solution was seeded whereupon crystallization started immediately. When distillation could no longer be effected on the steam bath, the mixture was allowed to cool then filtered and thoroughly washed with toluene. The cream colored fine

crystalline product was dried at 150°C for 16.5 hours. The recovery was 21.2 g, 98.5%. See Table 1 for properties.

Recrystallization from Acetone-Acetic Acid. A 25 g sample of the above once recrystallized product was dissolved in acetone, treated with Darco G-60 and distilled as described above. A volume of 150 ml of glacial acetic acid was added as described above instead of toluene. The first crop of product was removed by filtration and washed with 50 ml of acetone-acetic acid 1:1. On concentrating the mother liquor to a volume of about 100 ml on the hot plate, a second crop of about 1.5 g was obtained. The two crops were combined and stirred in 150 ml of 50% (vol.) solution of aqueous methanol. The slurry was filtered and the product was washed with 100 ml of methanol on the funnel and dried at 150°C for 16.5 hours. The product resembled that from acetone-toluene and the recovery was 23.3 g, 93%. See Table 1 for properties.

Recrystallization from Acetone-Acetonitrile. A solution of 25 g of the above once recrystallized product was made and treated as described above. To this was added 200 ml of acetonitrile and the solution was distilled with moderate stirring. When about 100 ml of distillate had been removed, an additional 200 ml of acetonitrile was added at about the same rate as that at which the distillate was removed. When the volume had reached about 125 ml and marked crystallization had occurred, the mixture was cooled to room temperature, filtered and the product was washed first with 100 ml of cold acetonitrile then with 100 ml of ether. After the glistening cream colored crystalline product was dried at 150°C for 16.5 hours, the recovery was 23.5 g, 94%. See Table 1 for properties.

Recrystallization from Acetone-Benzene. A 7 g sample of DODECA similar to the above once recrystallized product was dissolved in 100 ml of acetone, treated with 5 g of Darco G-60, filtered through a fiber glass filter and the carbon pad was washed with 35 ml of acetone. The amber colored solution was distilled as described above until 30 ml of distillate had been removed. Then as the distillation continued, 65 ml of benzene was added at about the same rate as that at which the distillate was removed. When a total of 75 ml of distillate had been removed, the solution was seeded and crystallization started immediately. When the temperature of the distillate reached 79°C, the distillation was discontinued, the mixture was cooled to room temperature and filtered. The product was washed thoroughly with benzene and dried at 150°C for 16.5 hours. The product was similar to that from acetone-toluene and the recovery was 6.2 g, 89%. See Table 1 for properties.

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2. Explosives - Synthesis
3. Explosives - DODECA

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HEAT RESISTANT EXPLOSIVES XXII. THE PREPARATION OF 2,2',2",2''',4,4',4",4''',6,6',6",6'''-DODECANITROQUATERPHENYL, DODECA (C), by J. C. Dacons. 28 Jan. 1966. v.p. BuWeps task RMMO 62 058 F008 08 11.

CONFIDENTIAL

The compound 2,2',2",2''',4,4',4",4''',6,6'6",6'''-dodecanitroquaterphenyl has been prepared in 70% yield by the treatment of 3-chloro-2,2'4,4'6,6'-hexanitrobiphenyl with copper bronze in nitrobenzene at 190°C. When purified by recrystallization from mixed solvents, the product was chromatographically pure and showed no tendency to melt at 450°C. (U)

1. Explosives, Heat resistant
2. Explosives - Synthesis
3. Explosives - DODECA

I. Title
II. Dacons, Joseph C.
III. Project

DOWNGRADED AT 3 YEAR INTERVALS DECLASSIFIED AFTER 12 YEARS

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